

PATENT SPECIFICATION

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COMPLETE SPECIFICATION.

A Vibration Damping Material Having a Broad Temperature Band Comprising Only One Copolymer.

We, FARBWERKE HOECHST AKTIENGESELLSCHAFT, vormals Meister Lucius & Brüning, a body corporate recognised under German law, of Frankfurt (M)-Hoechst, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to a vibration damping material having a broad temperature band comprising only one copolymer.

By vibration damping materials there are to be understood coverings of materials that when applied to sheet constructions, for example, damp their vibrations (resonance). In contradistinction to the materials already known that absorb the sounds transmitted by air, vibration damping materials reduce the sound vibrations in solids so that a radiation of sounds transmitted by air is avoided from the source.

Vibration damping materials have become more and more important in industry and increasing demands have been made on the quality of the damping coverings. Considerable efforts have been made to develop materials which ensure an effective vibration damping at extremely low and high temperatures as well as at normal temperatures, that is to say the temperature band (that is, range of temperature) of vibration damping at the customary frequencies of 100 to 200 cycles per second (Hertz) should be so broad that all demands of modern technical applications are met. A covering material that, when applied to metal or metal containing constructions, especially to sheet metal or sheet metal constructions, or to hard materials, for example hard plastics, uniformly

damps the vibration over a wide temperature range, is referred to in this Specification as a vibration damping material having a broad temperature band.

The vibration damping effect achieved is determined by measuring the mechanical loss factor d (cf. German Industrial Standards (DIN) 1332) of the combination of the hard material (for example sheet metal) and the covering. The temperature range over which a good vibration damping effect is obtained is defined by the temperatures at which the loss factor d of the combination has decreased to half the maximum value. A uniform vibration damping effect is obtained when the mechanical loss factor d within the total temperature range that is of interest does not fall below half the maximum value. Composite materials are of interest from a technical point of view in which this half-value of the loss factor d amounts to at least 1 per cent. (In this case the vibration damping of the combination corresponds approximately to the vibration damping of wood or cardboard). Such vibration damping material having a broad temperature band has, at the same time, the advantage of being effective within a wide frequency range at a given temperature.

The conditions that a vibration damping material must fulfil in order to attain as high a damping action as possible on, for example, sheet metal constructions and the methods of measurement are already known (cf. H Oberst, K. Frankenfeld, *Acustica* 2 (1952) AB 181; H Oberst, G. W. Becker, K. Frankenfeld, *Acustica* 4 (1954), 433). Usually, when high weight polymers have been subject to a vibratory stress, the vibration damping materials have made use of the maximum of the internal mechanical

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losses (hysteresis) occurring in the transition from the hard to the soft state (second order transition range). The temperature range of this maximum depends on the second order transition temperature of the substance and has usually been determined volumetrically. However, by the second order transition temperature there is to be understood in this Specification the temperature at which, in the short time measurement with the above mentioned reference frequency of 100 to 200 c.p.c., the maximum of mechanical losses occurs. This second order transition temperature defined in a short time experiment is, in general, about 30° to 50° C. above the volumetrically determined temperature. (Cf. H. A. Stuart "Die Physik der Hochpolymeren", published by Springer-Verlag, 1955, vol III, chapter X, especially par. 55 e 3, chapter XI, par. 58 and table XI, 2, page 667.)

All the hitherto known vibration damping materials have consisted of homopolymers or copolymers having a relatively homogeneous structure and which have exhibited a sharp transition between the hard and the soft state and, therefore, have possessed the disadvantage that the temperature range within which a good damping has been exhibited has been relatively limited.

The present invention provides a vibration damping material having a broad temperature band which comprises a single copolymer that has a chemically heterogeneous structure and is derived from at least two monomers, the second order transition temperatures of the homopolymers of which differ by at least 20° C.

Chemically heterogeneous copolymers of this kind containing adjacent polymer portions containing highly differing proportions of the incorporated monomers, are obtained either by using monomers having highly differing polymerization rates in, for example, a normal single-pot polymerization, or when using monomers having the same or similar polymerization rates by varying the ratio of monomers during the polymerization, for example by the process described in British Patent Specification No. 39820/60 (Serial No. 971,629).

It is known that in a chemically homogeneous copolymer, that is a copolymer in which the ratio of the monomers within the individual macromolecules in the copolymer remains practically constant, the second order transition temperature of the copolymer is situated between the second order transition temperatures of the homopolymers obtained from the monomers of which the copolymer is composed when the second order transition temperatures are different; the second order transition temperature of the copolymer and, correspondingly, the damping centre is shifted nearer to the range

of the lower temperatures the higher the content of the monomers whose homopolymer has the lower second order transition temperature. This effect is called "internal" plasticizing. The relation between the chemical composition of copolymers and their second order transition temperature is known for many copolymers (cf., for example, L. A. Wood: "Glass transition temperature of copolymers", J. Polym. Sci., 28 No. 117, (1958), p. 319; K. Schmieder and K. Wolf, Kolloid-Zeitschrift (1953), page 149 et seq.).

We have also found that the relation between the second order transition temperature and the ratio of monomers for the copolymer portions formed in one polymerization batch containing a composition of monomers that form a chemically heterogeneous copolymer is the same as for the chemically homogeneous copolymers formed from the same monomers. If, for example, in a polymerization process in which chemically heterogeneous copolymers are obtained, there is used a plasticizing monomer that polymerizes more rapidly than the other monomer or monomers, at the beginning of the polymerization there is obtained a polymer portion consisting almost entirely of the plasticizing monomer, so that the copolymer finally obtained by the polymerization exhibits a vibration damping in that range of temperature in which the second order transition temperature of the homopolymer of the plasticizing monomer is situated and in which, therefore, the homopolymer has its damping centre. Correspondingly, additional ranges of vibration damping are obtained in those temperature ranges which correspond to the second order transition temperature of the copolymer portions which form additionally and which have a different composition of monomers.

To prepare copolymers having as uniform a vibration damping effect as possible over a broad temperature range, that is copolymers for use in the present invention, the following possibilities exist:—

(1) The copolymerizable monomers are selected in such a manner that the second order transition temperatures of the homopolymers of the corresponding monomers (which temperatures are, in most cases, known from the pertinent literature) correspond to the limits of the desired temperature range of the vibration damping (for example, 0° C. and 50° C. in Example 1) or comprise this range;

(2) When the monomers have different polymerization rates and the second order transition temperatures of the corresponding homopolymers do not encompass too large a temperature range (20° to 100° C.), the desired heterogeneous copolymers, which effect a uniform vibration damping when

used as a covering, for example on sheet metal, can be obtained by the single-pot polymerization process. In order to ascertain the ratio of monomers that is most suitable for the desired purpose, some experimental batches containing different proportions of monomers, for example 20:80, 50:50 and 80:20, are polymerized in the single-pot process. Sheets having a thickness of 1 to 3 mm. are made from the copolymers obtained, applied to steel sheets or interposed between steel sheets, and the loss factor d of the combination is measured as a function of the temperature and the frequency in the desired ranges (usually 100 to 200 c.p.s.). By comparing and, if necessary, interpolating the different curves of vibration damping, the ratio of monomers is determined that will yield a copolymer having a uniform vibration damping effect in the desired range of temperature and frequency (cf. Example 1 below);

(3) When the monomers have different polymerization rates and the second order transition temperatures of the corresponding homopolymers differ considerably, separate damping centres at different temperatures (cf., for example, Fig. 3, curve 1) sometimes occur when the monomers are polymerised by the single-pot polymerization. In this case, and also when using monomers that have an approximately equal polymerization rate which, by the single-pot polymerization would yield homogeneous copolymers, copolymers suitable for use as vibration damping materials having a broad temperature band can be made by adjusting the monomer ratio during the course of the polymerization, for example as described in British Patent Specification No. 39820/60 (Serial No. 971,629).

From the second order transition temperatures of the homopolymers of the selected monomers, the second order transition temperature of the copolymer can be evaluated by linear interpolation in sufficient approximation by estimating the second order transition temperature proportionally to the molar composition of the two monomers. Single-pot batches each containing a different molar proportion of the monomers are polymerized, samples taken from time to time are measured with respect to the composition of the polymer and the measurements obtained are represented graphically in a conversion curve as a function of the reaction period. From the different copolymers thus obtained sheets are made having a thickness of 1 to 3 mm., applied to or inserted between steel sheets, and the loss factors d of the combinations are measured as a function of the temperature. By comparing the conversion and the vibration damping curves of the single-pot copolymers, the conversion curve for a copolymer

having the desired uniform vibration damping can easily be ascertained because of the dependence of the second order transition temperature on the composition of the copolymer portions. From the single-pot test copolymers it can be ascertained at which monomer ratio the desired copolymer composition corresponding to the conversion curve is formed, (cf. Example 2 in which a vibration damping material having a broad temperature band is made according to this method).

For the preparation of copolymers which may be used as vibration damping materials having a broad temperature band, there are advantageously used monomer mixtures consisting of at least one member of the following group (a) and at least one member of the group (b). Group (a) consists of monomers whose homopolymers have second order transition temperatures situated at or below room temperature, while the second order transition temperature of the homopolymers of the monomers of group (b) are situated at or above room temperature.

(a) Vinyl ethers, esters of alcohols having 4 to 12 carbon atoms with acrylic acid, methacrylic acid or maleic acid, vinyl esters of fatty acids having 4 to 12 carbon atoms, vinylidene chloride, butadiene and isobutylene.

(b) Vinyl esters of fatty acids having 2 or 3 carbon atoms, esters of alcohols having 1 to 3 carbon atoms with acrylic acid, methacrylic acid or maleic acid, acrylonitrile, vinyl chloride and styrene.

Thus copolymers are obtained from the above combinations that are effective at, below and above room temperatures and thus cover the most commonly required temperature range.

If, for special applications, vibration damping materials are required which only need to be effective at low or high temperatures, it is advantageous to copolymerize at least two monomers from only group (a) or from only group (b).

Preponderantly amorphous copolymers of ethylene and/or α -olefins having 3 to 10 carbon atoms and having an unbranched chain with members of group (b) are also suitable as vibration damping materials having a broad temperature band.

Monomers of which the homopolymers still possess, in addition to the main vibration damping range (range of second order transition temperature), a structurally conditioned secondary range of vibration damping are particularly suitable as the main component of the copolymers for use in the present invention, so that, as in the homopolymer, a vibration damping within an extensive range, although of a non-uniform height, is obtained. In order to obtain a vibration damping material having a broad temperature

band, it is only necessary to fill the vibration damping gap by a suitably controlled copolymerization, and, if necessary, to increase the secondary range of vibration damping. A co-monomer is advantageously chosen whose homopolymer has a second order transition temperature situated within the range of the secondary range of vibration damping of the main monomer. These particularly favorable conditions can be obtained with vinyl chloride copolymers because polyvinyl chloride has a broad and also a relatively high secondary range of vibration damping, (cf. Fig. 4, curve 2).

The copolymers used for the composition of vibration damping materials having a broad temperature band can be obtained by diverse polymerization processes, for example by suspension- or emulsion-polymerization. They may, for example, be used in the form of a dispersion or as a sheet to one side of sheet constructions of interposed between two or more sheets.

By the admixture with fillers which increases the modulus of elasticity of the copolymers the damping heights of the metal-covered-combination can be improved, particularly by fillers having a light specific gravity since, with the same mass ratio, a greater thickness of covering layer is obtained and the damping increases approximately quadratically with the ratio of the thickness. The dependence of the range of damping on the temperature is only influenced by a minor amount by the fillers. Suitable fillers are, for example, mica, expanded mica, graphite, carbon black, chemically pure silicic acid in the form of a sub-microscopically fine particles such, for example, marketed under the Registered Trade Mark "Aerosil", wood dust or cork powder. The ratio of the amounts of the copolymer and filler may vary within wide limits. Since the specific gravities of the individual fillers differ considerably from each another; the ratio of copolymer to the filler should preferably be from 1.5 : 1 to 0.5 : 1 in the case of light fillers, for example "Aerosil", expanded mica and carbon black; in the case of heavy fillers, for example mica, the ratio should preferably be from 1:8 to 1:2, the aforementioned figures being parts by weight.

By the addition of plasticizers, the range of vibration damping can be shifted to the range of lower temperatures. The plasticizer is preferably used in amounts ranging from 0 to 40 per cent by weight, advantageously 0 to 20 per cent by weight, calculated on the weight of the polymer. As plasticizers there may be used, for example, esters of phosphoric acid, preferably tricresyl phosphate, diphenyl cresyl phosphate trichlorethyl phosphate; esters of phthalic acid, preferably dibutyl phthalate, dioctyl phthalate or 2-ethyl-

hexyl-phthalate; diesters of phthalic acid and the monoethylether of ethylene glycol; esters of abietic acid, for example ethyl abietate; esters of adipic acid, for example benzyl butyl adipate or benzyl octyl adipate.

In the following examples a comparison is given between the different materials with a mass ratio of the covering to the sheet of 20% or 25%, respectively. In special cases, substantially thicker coverings may be used since they effect a better damping. However, owing to the increased load on the construction thicker coverings are, in most cases, impracticable from a technical point of view.

The following examples illustrate the invention, the percentages and parts being by weight unless otherwise stated:—

EXAMPLE 1.

In Fig. 2 of the accompanying drawings the mechanical loss factor d (damping d) is represented graphically as a function of the temperature for a number of coverings that were sprayed on sheet metal, the ratio of covering: plate being 20%. The measurements were made at a frequency of 200 c.p.s. by means of a bending resonance method (cf., for example, G. W. Becker, *Kolloid-Zeitschrift* 140 (1955, page 1, H. Oberst and K. Frankenfeld, *Acustica* 2 (1952), AB 181 and Robinson, *J. Sci. Instr.* 32 (1955) page 2). All the materials contained 8 parts of expanded mica per 10 parts of an aqueous starting dispersion of 55% strength, that is 60% of filler, calculated on the mixture of solids.

Curve 1 in Fig. 2 represents the damping curve of a broad temperature band vibration damping material having a moderate band width (half-value width about 50° C. to 60° C.). The damping material consisted of a chemically heterogeneous copolymer prepared from 63% of vinyl acetate (the homopolymer of which has a high second order transition temperature; cf. damping curve 2 in Fig 2) and 37% of maleic acid diester (the homopolymer of which could not be prepared in a pure state; the second order transition temperature is probably at about 0° C.), which was prepared by the single-pot emulsion process.

The importance of the choice of the monomer ratio is shown by the copolymer represented, for the purpose of comparison, as curve 3 in Fig. 2, this copolymer having been prepared by the single-pot process from vinyl acetate and maleic acid diester in a ratio of 77:23, respectively. This material only had a half-value width of damping d on sheet metal of about 35° C., and was, therefore, practically a temperature "narrow band" absorber. The most suitable monomer ratio can be ascertained by a comparison of the d -curves.

Curve 4 of Fig. 2 represents the damping d of a copolymer prepared from 63% of vinyl acetate and 37% of maleic acid diester, that is a copolymer corresponding to curve 1, which copolymer was additionally plasticized with 5%, calculated on the polymer, of dibutyl phthalate. The temperature range in which a good damping effect was obtained was shifted to the range of lower temperatures with a simultaneous extension. The advantage of the plasticizing, in this case, lay in fact that the damping was satisfactory at temperatures down to about 0° C.

The damping height of more than 10%, which was very high as compared with the vibration damping material containing no fillers and described in Example 2, was due to two causes. The somewhat lower temperature band width resulted on the one hand, in higher absolute values of the damping, and, on the other hand, the polymer became lighter owing to the filling with the expanded mica which has a lighter specific weight. With the same amount of copolymer, a higher thickness of covering could be obtained whereby the damping effect was increased considerably.

EXAMPLE 2.

Curve 1 in Fig. 1 represents the change in the copolymer composition with the increasing conversion, expressed as % of the total conversion (conversion curve) for a single-pot copolymer of vinyl chloride and 2-ethylhexyl-acrylate (octylacrylate; briefly referred to hereunder as OA) with a ratio of the monomers VC:OA=85:15. A pure OA homopolymer had a second order transition temperature of about -40° C., while PVC had a second order transition temperature of +100° C.

Owing to the higher polymerization rate of the plasticizing monomer OA, the polymer portions which formed at first had a higher content of OA (highest content 50%) and a lower second order transition temperature. In the course of the polymerization the OA was consumed and the polymer structure changed relatively quickly into portions containing a higher percentage of VC and having a high second order transition temperature. The vibration (resonance) damping effect of this material on sheet metal, that is the mechanical loss factor d of a combination with the sheet metal in the case of a mass ratio covering: plate of 25% and at a frequency of 100 c.p.s., is represented as a function of the temperature in curve 1 of Fig. 3. The damping was not distributed uniformly over the temperature range. The weak damping range at 40° C. is also exhibited by pure PVC ("secondary damping range") and is, therefore, proportional to the vinyl chloride proportion of the copolymer; the further path above -40°

C. depends on the structure of those proportions of the copolymer that had a higher or lower content of acrylate and different second order transition temperatures corresponding to the conversion curve 1 of Fig. 1.

A copolymer that could be used according to the invention was obtained by a suspension polymerization using 15% of OA according to the conversion curve represented by curve 2 of Fig. 1; the copolymer exhibited a uniform distribution of damping d on sheet metal as represented in curve 2 of Fig. 3 (mass ratio covering: plate=25%; frequency 100 c.p.s.). This chemically heterogeneous copolymer was prepared according to British Specification No. 39820/60 (Serial No. 971,629); a monomer mixture of 15 parts of OA and 30 parts VC was placed in the polymerization vessel (ratio OA:VC=1:2; cf. conversion curve 3, Fig. 1) and after one hour (point A) the remaining 55 parts of VC were continuously metered into the polymerization zone. The completion of this operation is shown at point B.

The total polymer according to Curve 2, Fig. 1, contained, as compared with the single-pot polymer, a proportion having a particularly high content of acrylate (highest content 78% OA), a smaller proportion having a medium OA content and a higher proportion having a high VC content, that is a high second order transition temperature. This resulted in a more effective damping within the range of -40° C. to +20° C. (cf. damping curve 2 of Fig. 3) which was due to the proportion having a higher content of OA, that is a lower second order transition temperature, a diminution of the damping within the range of +20° C. to +50° C. (due to the smaller proportion having a medium content of acrylate), and a more effective damping above +50° C. (due to the higher proportion of copolymer having a high content of VC with a high second order transition temperature).

1.5% of dibutyl tin mercaptide and 0.5% of calcium stearate were added to the particular copolymer described in this example, and the resulting mixture was processed into a calendered sheet having a thickness of 1.5 mm. and applied to a steel sheet having a thickness of 1 mm. (mass ratio covering: plate=0.25). Curve 2 of Fig. 3 represents the damping of this combination of covering + plate as a function of the temperature (measured by means of the bending resonance method).

When applying the same amount of covering material, the absolute damping height of a vibration damping material having a broad temperature band must, of necessity, be lower than in the case of a narrow band absorber which is only effective

within a temperature range of 30° C. while d may have a value up to 10%. For the purpose of comparison it is mentioned that in the case of cardboard and wood d is of the order of magnitude = 1%.

EXAMPLES 3 and 4.

Examples 3 and 4 (cf. the respective curves 1 of Fig. 4) show the d -curves (measured as described above) of two vibration damping materials having a broad temperature band prepared from 2-ethyl-hexyl-acrylate (OA) and vinyl chloride (VC) in a normal single-pot suspension polymerization process. In Example 3, VC and OA were placed in the polymerization vessel in the monomer ratio of 90:10, while in Example 4 this ratio was 70:30. The latter copolymer was, moreover, admixed with 33 parts of carbon black.

All the curves represented in Fig. 4 (a) and (b), show the damping d of sheet covering combinations at a frequency of 100 c.p.s. for a mass ratio covering : sheet = 25% as a function of the temperature.

These two products can be employed on an industrial scale in moderately broad temperature ranges ΔT , of 60° C. to 80° C.

In Example 3, Fig. 4(a), the most effective damping range was situated at temperatures within the range of 30° C. to 90° C. which is of importance, for example, for the vibration (resonance) damping of crankshaft and machine housings that have working temperatures above room temperature. The damping curve of pure PVC represented in Fig. 4(a), curve 2, illustrates the great advantage of the heterogeneous copolymer even when it contained only 10% of the second monomer as compared with a chemically homogeneous covering material, for example polyvinylchloride. The weaker damping range occurring below 0° C. was always obtained when using copolymers containing vinyl chloride (structurally conditioned "secondary damping range") and constituted a special advantage of vibration damping materials containing vinyl chloride.

The vibration damping material according to Example 4, Fig. 4(b) containing higher content of acrylate was very advantageous from a technical point of view in spite of the somewhat non-uniform damping distribution because the damping centre was situated precisely at the main working temperatures of about 20° C. whereas good damping properties were also exhibited in other, especially in higher, temperature ranges. For the purpose of comparison, a preponderantly homogeneous vibration damping covering usually employed in industry (copolymer vinyl acetate/maleic acid diester in the ratio of 77:23 containing 8% of dibutyl phthalate), the efficacy of which, especially after high temperatures, declines rapidly is represented in Fig. 4(b), curve 2.

The vibration damping material consisting of a heterogeneous copolymer according to Example 4 was of particular value when employed, for example, for the vibration (resonance) damping of automobile bodies or rail-road carriages since its efficacy was not only guaranteed in the temperature range of 10° C. to 30° C., that is in the range of the average annual temperature, but also to a sufficient degree when subjected to strong solar radiation or frost, or, for example, when used for washing machines which may attain a temperature of up to 60° C. in their "hot" operational phase.

WHAT WE CLAIM IS:—

1. A combination of one or more resilient members capable of undergoing vibration by periodic deformation with a vibration damping material, the said material comprising a copolymer having a chemically heterogeneous composition obtained by copolymerising two or more monomers, the second order transition temperatures of the homopolymers of which differ by at least 20° C.
2. A combination as claimed in Claim 1, wherein one of the monomers is vinyl chloride.
3. A combination as claimed in Claim 1, wherein the copolymer is obtained by the process described in British Patent Specification No. 39820/60 (Serial No. 971,629).
4. A combination as claimed in Claim 1, wherein at least one of the monomers is selected from the group consisting of vinyl ethers, esters of alcohols having 4 to 12 carbon atoms with aliphatic olefinically unsaturated carboxylic acids having 3 or 4 carbon atoms, vinyl esters of fatty acids having 4 to 12 carbon atoms, vinylidene chloride, butadiene and isobutylene.
5. A combination as claimed in Claim 1 or Claim 4, wherein at least one of the monomers is selected from the group consisting of vinyl esters of fatty acids having 2 or 3 carbon atoms, esters of alcohols having 1 to 3 carbon atoms with aliphatic olefinically unsaturated carboxylic acids having 3 or 4 carbon atoms, acrylonitrile, vinyl chloride and styrene.
6. A combination as claimed in any one of Claims 1 to 5, wherein the vibration damping material also contains a filler.
7. A combination as claimed in any one of Claims 1 to 6, wherein the vibration damping material also contains up to 40% by weight of a plasticiser, calculated on the weight of the copolymer.
8. A combination as claimed in Claim 7, wherein the vibration damping material contains up to 20% by weight of the plasticiser, calculated on the weight of the copolymer.
9. A combination of one or more resilient members capable of undergoing vibra-

tion by periodic deformation with a vibration damping material, the said material comprising a copolymer having a chemically heterogeneous composition obtained by copolymerising two or more monomers, the second order transition temperatures of the homopolymers of which differ by at least 20° C., and the said material being substantially as described in any one of Examples 1 to 4 herein.

10 1 to 4 herein.

10. A combination as claimed in any one

of Claims 1 to 9, wherein the resilient member comprises a metal plate.

11. A combination as claimed in any one of Claims 1 to 9, which comprises a pair of metal plates separated by the said vibration damping material.

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COMPLETE SPECIFICATION

3 SHEETS

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Sheet 1

FIG. 1

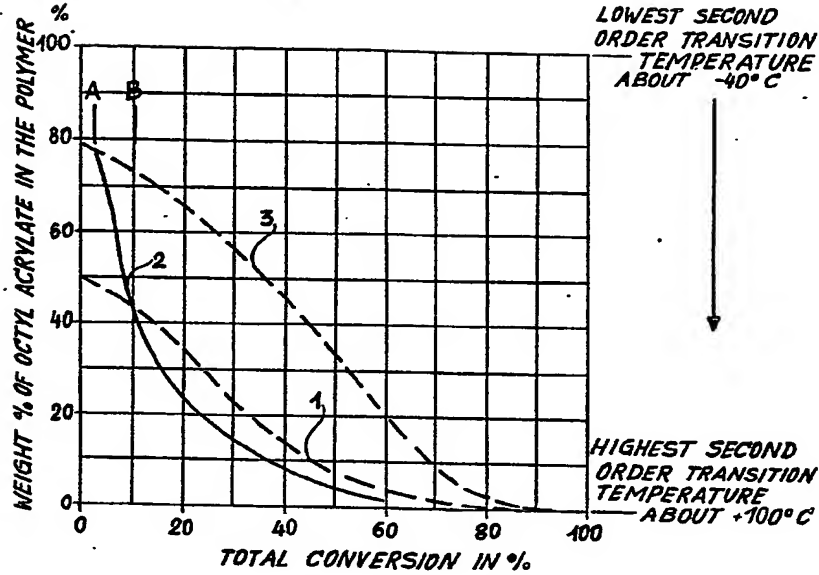


FIG. 2

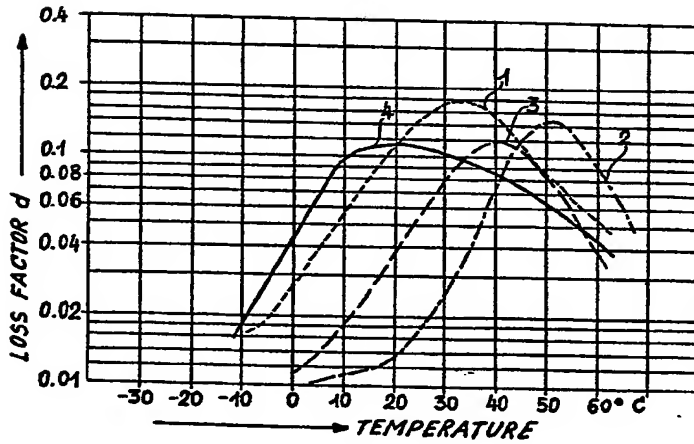
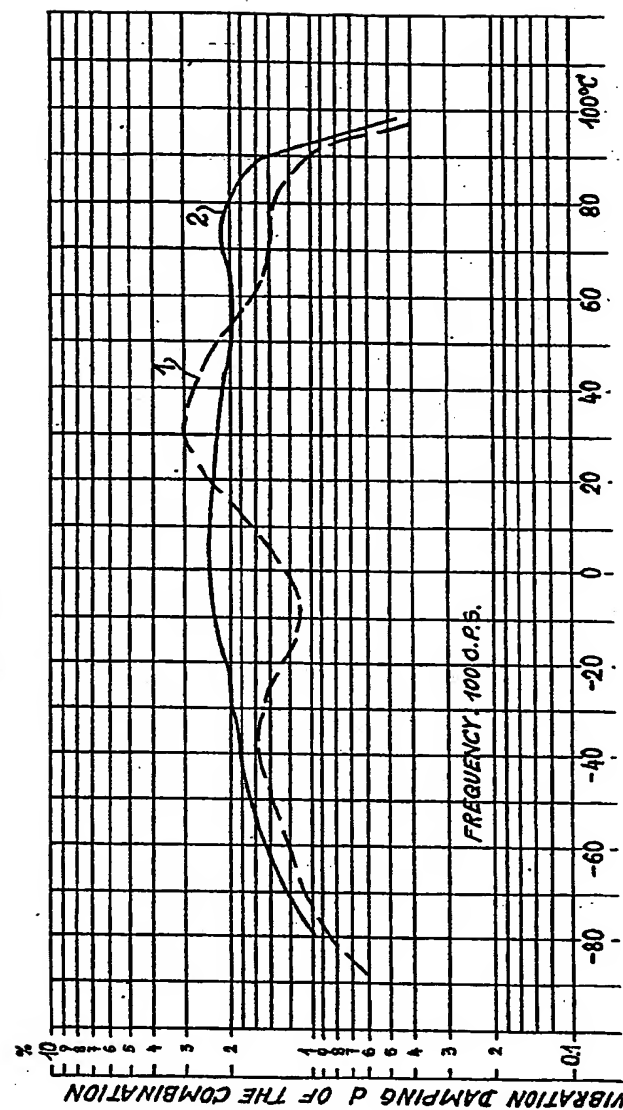


FIG. 3



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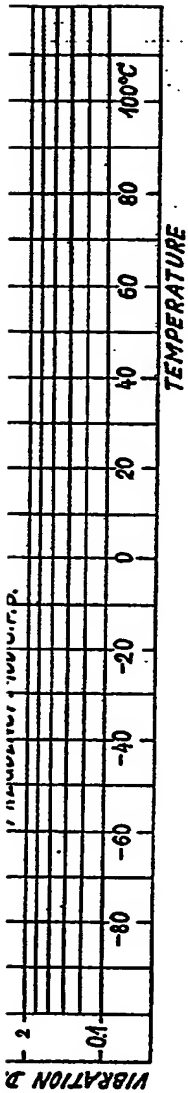
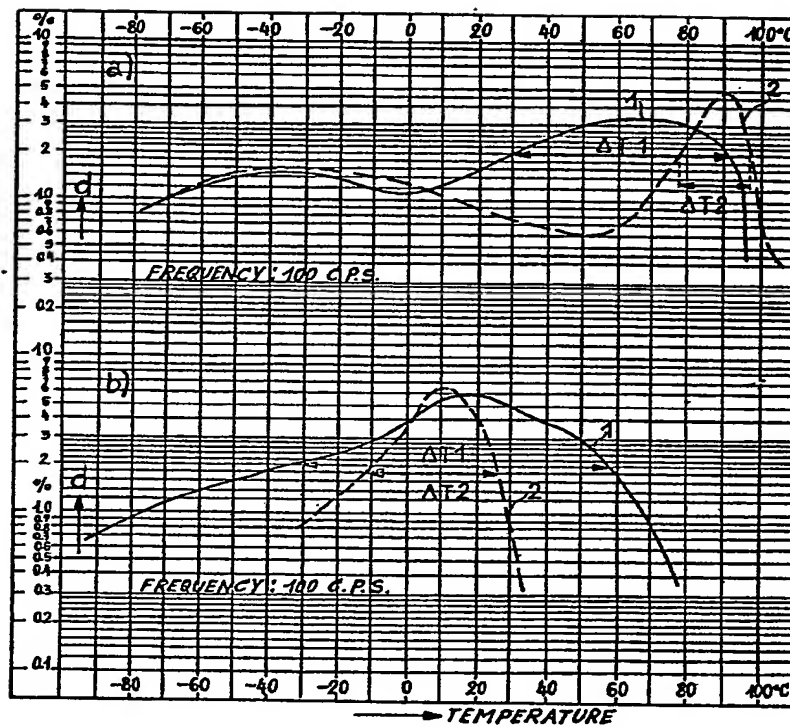
COMPLETE SPECIFICATION

3 SHEETS

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Sheets 2 & 3

FIG. 4



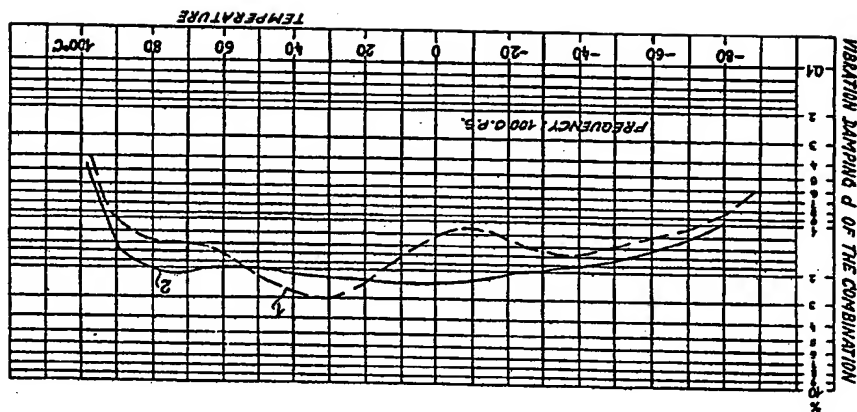
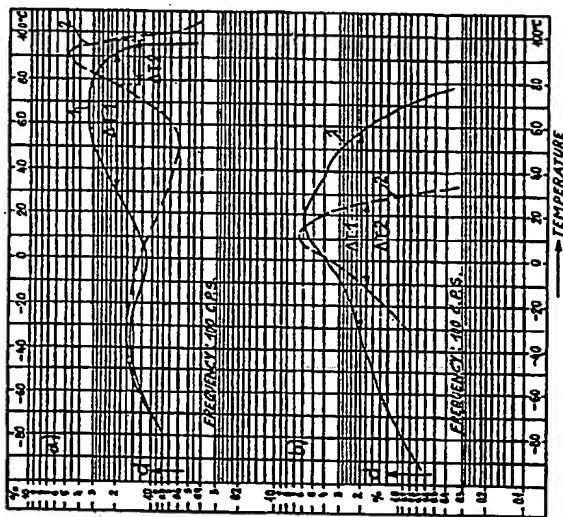


FIG. 3

FIG. 4



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